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Clean and Hazardous Chemicals Laboratory at
National Water Research Institute, Canada
Centre for Inland Waters

B. K. Afghan and J. Lawrence

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CLEAN AND HAZARDOUS CHEMICALS LABORATORY

AT

NATIONAL WATER RESEARCH INSTITUTE

CANADA CENTRE FOR INLAND WATERS

by

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Environmental monitoring for hazardous chemical contaminants produces a constant demand for improved and more sensitive analytical methodologies for quantitative analysis and confirmation at ultra-trace levels (pico-gram or sub-nano gram levels). For example, 2,3,7,8-tetrachloro dibenzo-p-dioxin has recently been recognized as probably one of the most toxic chemical contaminants known to man and a reliable methodology is needed to measure environmental concentrations and assess its toxicity and impact.

Significant errors can arise in analysis of contaminants present at low parts per trillion levels for several reasons. Particulate matter which has a tendency to adsorb organics from the laboratory atmosphere can contaminate samples during processing. Many synthetic materials used in the construction of laboratories contain leachable, volatile organics, such as phthalate esters, and these can cause difficulties during quantitation. In addition, the safety of laboratory personnel could be jeopardized when handling hazardous chemicals and/or developing methodologies for hazardous substances. Various government agencies are becoming increasingly concerned with the use of known and potentially hazardous, carcinogenic and highly toxic substances in their laboratories. At the same time, these agencies are increasing their efforts to ensure that good quality data are produced so that baseline studies and assessment of various hazardous chemicals can be carried out.

In 1978 to 1979, a need was recognized within the Analytical Methods Division (AMD) of the National Water Research Institute for a special laboratory facility, to carry out methods development research on hazardous compounds (eg, 2,3,7,8-TCDD and nitrosamines) at ultra-trace levels. The primary aim was to provide maximum protection for the laboratory staff and other personnel in the building as well as an isolated clean environment to minimise cross-contamination of samples during concentration and cleanup steps. Although the laboratory was primarily intended for analytical methods development and confirmation, it was designed as a multi-user facility to accommodate the requirements of several programs at CCIW.

This article will describe the special features of the laboratory, the compromises that had to be made, some of the difficulties encountered during the construction, the special safety procedures that will be adopted and the analytical instrumentation and research that will be carried out in this laboratory. The laboratory contains features from clean rooms used by chemists for ultra-trace analysis combined with some protective containment features used by biologists when working with contagious organisms.

Laboratory Design

The primary consideration in the design was to build a versatile facility with high safety standards to carry out methods

development work for organics at ultra-trace levels. The general design criteria for the above were considered to be:

- 1) All air entering the laboratory must have negligible particulate level and should not contain any volatile organics.
- 2) All the walls, ceiling, windows and fixtures must be as airtight as possible to prevent dust penetration.
- 3) All the walls, ceilings and floor coverings must be of nonshedding and unleachable materials to minimize the particulate level and to eliminate leaching of chemical vapours into the atmosphere.
- 4) The laboratory must be kept under constant negative pressure. The negative pressure must increase within various zones in the laboratory and should be maximum in the area where toxic chemicals are to be stored.
- 5) The laboratory must be equipped with well designed hoods and enclosed work areas where sample concentration, cleanup and pretreatment can be carried out without exposing workers to any potential health hazards as well as minimizing sample cross-contamination.

- 6) The air leaving the laboratory must undergo decontamination prior to its exhaust into the open atmosphere.
- 7) The instrumentation must be compact, easily serviceable and easy to decontaminate in case of any hazardous chemical spills.
- 8) The laboratory must be isolated from all other laboratories in the building.

A floor plan of the laboratory is shown in Figure 1. The laboratory is divided into five areas which are arranged in a dead end chain with each area separated by a door. The air pressure increases negatively with respect to the rest of the building as one moves from Zone 1 to the glove box in Zone 3 (as shown in Figure 2). An electrical interlocking system is used on the doors between the entrance vestibule/zone I and zone II/shower/zone III so that both doors cannot be opened simultaneously. This ensures that a clean (low particulate) atmosphere is maintained and it also improves safety within various areas of the laboratory.

The entrance vestibule is used as a hand washing station, as a change room for putting on disposable lint free clothing, and for storage of samples for analysis. A desk is provided for workup of

data generated within the laboratory. A log is maintained of all persons entering the lab as well as of the number of samples and the type/quantity of hazardous chemicals used in the facility. The vestibule also accomodates a service panel for all electrical services within the laboratory.

Zone I is designated as the main area for pretreatment of samples as well as for the experimental work for concentration, cleanup and derivation prior to analysis. The majority of experimental work during methods development and routine analysis will be carried out in this area. The area is equipped with 2 L-shaped enclosed work areas for sample preparation and clean-up. One of the enclosed work areas is divided by a vertical sliding sash to allow the weighing of chemicals in an isolated environment. These work areas and the inside wall surface are stainless steel. They are equipped with sinks, electrical and gas services with remote controls and independent exhaust. Special ventilated storage cabinets for chemicals and solvents are also provided. Each work area is provided with horizontally sliding glass doors (3'x3') which allows an average face velocity of 150 fpm for a single opening. The doors are connected with an automated alarm system which is activated when more than one door is opened at any time. This ensures that the optimum face velocity is maintained in order to retain volatile chemical vapors and minimize out-leakage of fumes. The exhaust air is HEPA filtered.

Zone I is also equipped with two additional hoods; (i) an acid hood specially designed to provide horizontal as well as vertical air flow to eliminate any build up of acid fumes and again to protect the operator during experimentation. The acid hood has an independent air supply which is filtered through activated carbon and HEPA-filter. (ii) A modified class II hood provides an ultra-clean (negligible particulate matter) atmosphere for carrying out experiments where samples must be exposed to the open atmosphere for a considerable length of time. The clean air from the laboratory enters the modified class II hood and is recirculated through several HEPA-filters in series prior to entering the work area of the hood. The face of the hood is also provided with an air curtain which acts as a barrier between the working area within the hood and the environment of the laboratory. Again this design is to protect both the sample and the operator from contamination. The exhaust from both hoods is HEPA-filtered prior to discharge to the open atmosphere. These hoods are also provided with an independent and remotely controlled water supply as well as electrical and gas services. Zone I has sufficient stainless steel bench top space to accommodate ovens, furnaces and other general purpose instrumentation. A cartridge water deionization system is installed to provide high purity water in this zone of the laboratory.

Zone II is designed as an instrumentation room. High pressure liquid chromatography and gas chromatography are the major

techniques which will be employed during the optimization of methodology and the screening of samples for ultra-trace analysis. These samples will be further confirmed using other techniques such as GC/MS which is located in another laboratory on the same floor. A flexible elephant trunk exhaust system is installed above the bench areas to provide the necessary exhaust for solvent and other gaseous vapours generated by the analytical instrumentation. The benches are also provided with enamelled cabinets for storage of spare parts and other consumable items. This zone is equipped with a modified ultra-pure clean air hood (class II) to be used for storing the samples to be analysed as well as to serve as a link to zone III where the hazardous substances will be used and stored. The modified class II hood is connected via a "pass-through chamber" with the glove box in zone III. The pass through has two doors which are electrically interlocked so that both doors can not be opened at any given time. All the samples that need spiking, vials for dilute standards, etc., that will be required in Zone III, will be placed in a glass tray and left in the pass-through. These will then be picked up by a person in zone III.

Similarly, all standard solutions prepared in this glove box will be brought to zone I or II via the "pass through chamber" and the modified class II hood. The reason for this particular design and practice was to limit the handling of concentrated solutions of hazardous chemicals in other zones of the laboratory. This area would

also serve to localize any solutions containing hazardous materials so that in case of accident the area can easily be decontaminated.

Zone III is an L-shaped room and contains a change room/shower area as well as a specially designed stainless steel glove box which is used to store the hazardous substances and concentrated solutions of toxic chemicals. The change room/shower area is separated from zone II and the glove box area by two electrically interlocking doors. All persons entering zone III would be forced to leave all samples and/or vials in the pass-through chamber attached to the modified class II hood in zone II. The operator would then dress in a disposable gown before working in the glove box. A shower is also provided in the change room for washup or decontamination.

The containment facility for the hazardous substances has several sections; (i) weighing area and storage area for solid materials, (ii) dilution and work up area, (iii) pass-through chamber, and (iv) modified class II hood. All are arranged to form a single unit. Weighing and work up area is a divided 6-port glove box. This part of the containment facility is located in zone III. The glove box area is kept constantly at a negative pressure of 0.5" water with respect to the surrounding area of zone III. The two individual compartments are separated by a vertically sliding sash. The area at the end of the glove box will be used for storing, weighing and preparing the stock solution of hazardous substances. The area nearer

the pass through will be used to spike the samples or prepare dilute standards. The pass through is the only entrance to the glove-compartment. All work will be carried out using the glove-ports.

The laboratory includes several additional features, such as stainless steel bench tops and enamelled storage cabinets, epoxy sealed surfaces, seamless flooring with seamless floor to wall interface regions, eye-wash, "hands-free" intercom system that permits voice communication, fire alarm systems, monitoring systems for air flows and measuring negative pressures of various zones within the laboratory, back-up battery-operated emergency lighting, and anti-static circuitry. In addition, floor corners have been curved and dust-trapping ledges eliminated for ease of cleaning. Viewing windows and angled mirrors are provided so that workers in the laboratory can be observed from the outside.

Airflow and Filtration

In order to decrease airborne particulate matter and minimize buildup of volatile vapors in the clean rooms, it is important to have laminar airflow so that all air-volumes move through the various areas with uniform velocity. The laboratory must be supplied with sufficient air input to provide proper movement through the laboratory as well as through the hood exhausts. To

maintain the lammer flow pattern it is important that air must not recirculate within the room. The laboratory has an independent air supply which is carbon and HEPA-filtered prior to entering the laboratory. The outgoing air is HEPA-filtered to remove any chemical dust or particulate matter generated within the laboratory before being exhausted to the outside of the building. The only exception is the acid hood in Zone I. The exhaust from these cabinets is not filtered because derivation work is done with dilute solutions and no particles are generated during the work up of samples.

The laboratory is designed to provide varying airchanges ranging from one air change in every 7 minutes in the entrance vestibule area to one airchange per $1\frac{1}{2}$ minutes in zone III.

The existing air supply to the building has a 2 stage dry type filtration system with a final filtration efficiency of 85% NBS dust spot. The Clean and Hazardous chemicals laboratory required a new facility where the existing building supply air was passed through activated charcoal and HEPA filters to remove volatile atmospheric organics as well as airborne particulate matter to negligible levels. The laboratory also required large exhaust air volumes (and thus supply air volumes) in order to maintain negative pressure patterns within the room and to provide the necessary ventilation. The following were determined as optimum exhaust ventillation requirements for various areas within the CHCL:

- Entrance vestibule exhaust (50 CFM)
- Exhaust for ventilated glass fume cabinet with horizontal modular 3'x3' sliding doors (1350 CFM)
- Exhaust for acid hood (1200 CFM)
- Class II hoods (2x480 CFM)
- Flexible elephant trunk exhaust system (140 CFM)
- Shower exhaust (100 CFM)
- Custom designed glove box (50 CFM), and
- General laboratory exhaust (300 CFM exhaust based on approximately 40 air changes)

The design of the air handling and purification system was very complex. A schematic of the air movement system is shown in Figure 2. A number of exhaust ventilation systems were combined to facilitate installation. The following combinations were found to be compatible and consequently were selected:

- Class II hood exhaust system: combined for Class II hoods in zone 1 and 2
- L-shaped cabinets in zone I are combined with entrance vestibule exhaust
- Acid fumehood exhaust
- Zone 3 general laboratory exhaust combined with shower exhaust and glove box exhaust.

All systems exhaust fans are located in the mechanical penthouse above the CHCL. The booster fans provide supply air at the required quantities in the CHCL. These are located above the ceiling in the corridor outside the laboratory.

Hoods and Enclosed Work Areas

Ventilated hoods and enclosed work areas were required which would have sufficient exhaust to capture and retain the atmospheric contaminants generated within it. It was also decided that the hoods in CHCL should have sufficient face openings so that sample handling could be carried out easily. For example, it was decided to maintain 3'x3' opening in the area where sample concentration and cleanup will be carried out. This opening must have a face velocity of 150 fpm to eliminate outleakage of any hazardous vapours during experimentation. The acid hood and class II hoods in zone 1 and 2 needed extensive modification to satisfy the criteria for the CHCL. The containment facility (glove box, pass-through and Class II hood) for storing the hazardous chemicals had to be specially designed. The performance of this facility is constantly monitored. An auxiliary mechanism is included in case of power failure, change in air pressure or in the case of an accident. This mechanism will isolate the glove box area from the CHCL as well as from the building.

Electrical, Laboratory Plumbing and Other Services

Electrical services to the laboratory enter various areas by means of conduits. Seals are installed in and around the conduits to prevent cross contamination. The electrical distribution is achieved by 120/208 V 30A panel. This panel provides the entire power requirements of the laboratory including all outlets and lighting. A separate feed from the emergency power system of the building is also connected to this panel in order that the laboratory can operate should a power failure occur.

The lighting in the laboratory consists of double 4 ft fluorescent, glass lens type, surface mounted fixtures directly secured to the ceiling. Seals are installed in and around the conduits that enter the fixture from the ceiling cavity above to prevent dust leakage. The light fixtures are finished in white baked enamel. The light levels for general illumination are between 50 and 80 foot candles. Supplementary lighting for enclosed work areas and hoods is provided in the form of yellow light to minimize any photo-decomposition of the analyte during sample processing. These areas are also provided with UV-lights which will be used for decontamination in case of spill and/or accident. Light switches with silicon sealed stainless steel plates are used to control lights within the room.

An intercom system is installed to permit voice communication between the zoned areas. One station per zone and one for the change room/shower area is provided. The system operates "hands-free" once the call has been initiated. Alternatively, the system can be left with all zones "ON" for total "hands-free" operation. A telephone in zone I is also provided with extensions in zone II and III for external communication.

A fire alarm consisting of a smoke detector system and various other alarm systems are installed within the room and will be activated in case of fire, malfunctioning of airflow system or change in air pressure and air flow.

Standard plumbing services are provided and are detailed in the architectural drawings. All the gases needed in the laboratories are stored in the penthouse above the laboratory. A suitable manifold is used to deliver the necessary gases to the various zones within the laboratory. The flow of gases is controlled by two stage regulators outside and inside the laboratory.

Safety Program

The special safety features incorporated into the laboratory have already been described in the earlier sections. The laboratory

will be operated under the guidance of a multi-agency safety committee, consisting of the CCIW safety officer and representatives from NWRI, CCIW, Department of National Health and Welfare, and National Research Council. This committee will be responsible for compiling a special safety procedures manual for the laboratory and for ensuring day to day compliance by all staff using the facility. With the laboratory only having been opened a few weeks, it is too early to report on any specific safety problems encountered. However, our approach to safety will tend towards a common-sense appreciation of hazard: we will emphasize that the practice of safety in the chemical laboratory requires a desire on the part of the individual to protect himself and his associates as well as a need to follow a set of rules.

Chemical Monitoring and Medical Surveillance

To ensure a clean and safe working environment in the CHCL, the atmosphere will be regularly monitored for particulate matter and volatile organics. The detailed methodology for monitoring volatile contaminants in the laboratory will depend upon the type of work to be carried out. Bench tops, walls, etc. will also be monitored for possible spills or accidents. Appropriate decontamination procedures will be established depending upon the type of contamination detected.

The staff working in this laboratory will undergo extensive and regular medical surveillance. The health inventory examination for workers will include:

- Complete routine physical examination
- Genetic history
- Clinical laboratory profile
- Evidence of acute, subacute and chronic intoxication.

Role of Clean and Hazardous Chemicals Laboratory

The CHCL will be used to develop and document reliable analytical methodologies, including sample preparation procedures, for dioxins and other trace or hazardous contaminants. These documented methods will be distributed to analytical service laboratories which will be responsible for routine sample preparation and analysis. After methodologies have been developed, regular service laboratory facilities should be adequate for most routine sample processing. Stringent cleanliness will be required in the CHCL and therefore there will be some limit on the number of personnel working at any given time in the laboratory.

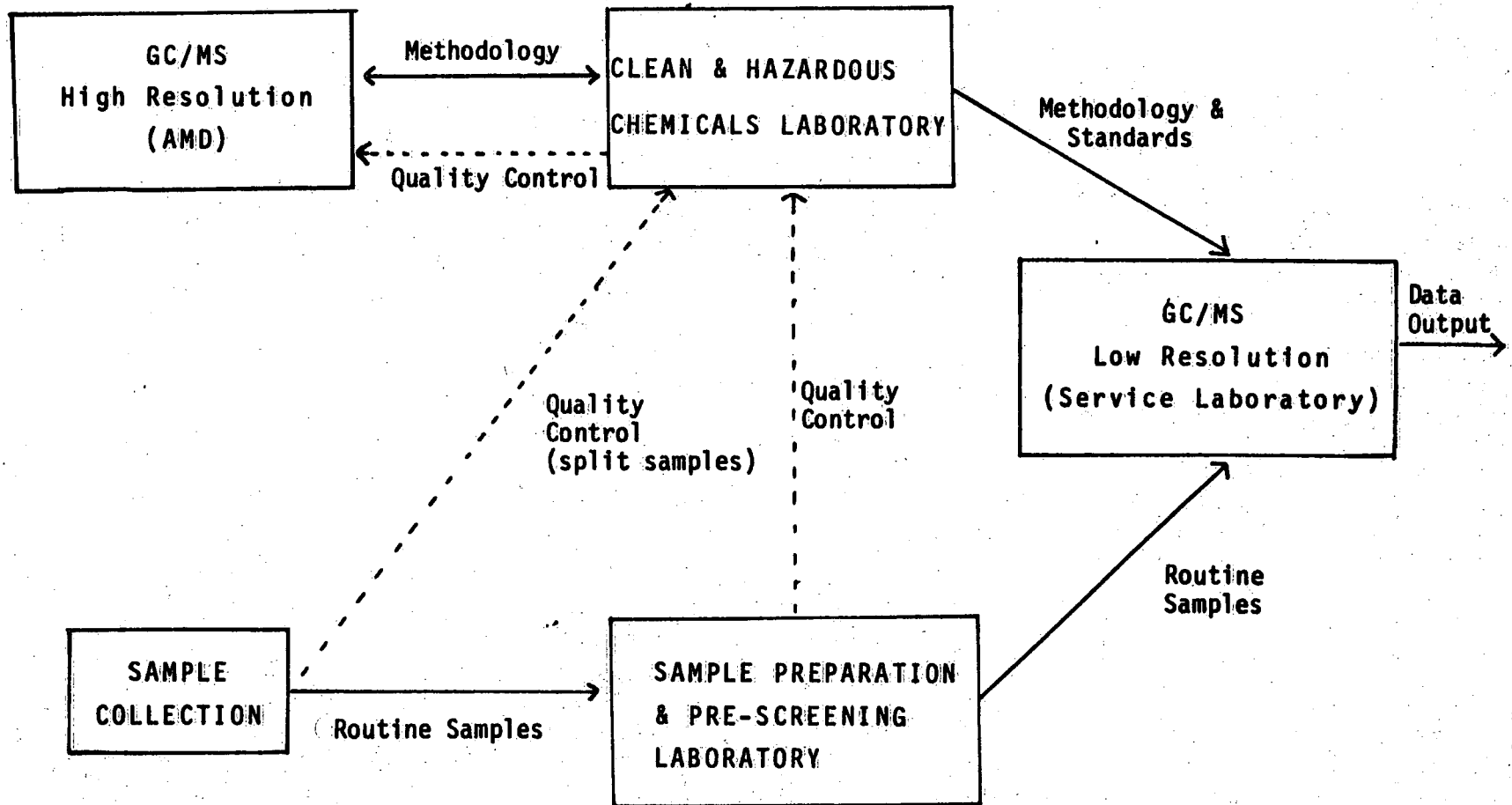
The role of the CHCL, once the methods have been developed, would be to prepare and provide diluted standards to the analytical service laboratories, provide confirmation service and to process and

analyze a limited number of split samples for quality control purposes. With this arrangement, routine sample processing and analysis can be carried out in the service laboratories, the standards and quality control will be of the highest calibre and the unique facilities of the CHCL can be used mostly for methods development or other research.

A diagram of the intended use of the laboratory is shown below:

ROLE OF CLEAN & HAZARDOUS CHEMICALS LABORATORY

IN ANALYSIS OF HAZARDOUS AND TRACE CONTAMINANTS



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FIGURES TO LEGEND

Figure 1 Floor plan of Clean & Hazardous Chemicals Laboratory

Figure 2 Schematic of air handling system for Clean & Hazardous Chemicals Laboratory

